Comments

The use of ethyl alcohol as an internal standard makes it possible to greatly simplify the procedure the quantitative determination of volatile compounds and methanol in alcoholic products. Before implementing a new method in a research laboratory, the method must be validated. Validation of the new method may be done based on experimental data obtained in the laboratory during testing of alcoholic products.

The use of the proposed method ensures high reliability of the data obtained, significantly reduces time, labor, material and financial costs. Analysis of volatile compounds in spirit drinks has never been so easy.

Here you can read modified text of official method, which allows to carry out analysis of alcoholic beverages usinf the developed method.

The places in the text document to be deleted are highlighted in yellow. Embedded parts of the test are highlighted in green. Highlighted in blue are data that will be established based on the results of the performed interlaboratory study.

National Standards Of People's Republic of China

GB/T 10345-2022
Instead of 10345-2007

GB/T 10345-2022

Analysis method of liquor

Method of Analysis for baijiu

Released on 2022-07·11

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Contents

Foreword

This document is drafted in accordance with the provisions of GB/T 1.1-2020 "Guidelines for Standardization Work Part 1: Structure and Drafting Rules of Standardization Documents".

This document replaces GB/T 10345-2007 "Analysis Methods of Liquor". Compared with GB/T 10345-2007, the main technical changes are as follows:

a) The method for determination of alcohol content is deleted (see Chapter 6 of the 2007 edition);
b) Deleted the determination method of total acid (see Chapter 7 of the 2007 edition);
c) the method for the determination of the total amount of esters has been added (see Chapter 8);
d) Added the determination methods of acetic acid and caproic acid (see Chapter 11, Chapter 12);
e) Changed the determination method of ethyl acetate, ethyl butyrate, ethyl hexanoate, ethyl lactate, n-propanol, β-phenethyl alcohol (see Chapter 10, Chapter 10, Chapter 13, 2007 edition, Chapter 11, Chapter 12, Chapter 15, Chapter 16);
f) Changed the determination method of ethyl propionate, diethyl acid (pimelic acid, suberic acid, azelaic acid) diethyl ester (see Chapter 13, Chapter 14, Chapter 14, Chapter 18 of the 2007 edition chapter);
g) The method for the determination of 3-methylthiopropanol (see Chapter 17 of the 2007 edition) was deleted.

Please note that some content of this document may be patented. The issuing agency of this document assumes no responsibility for identifying patents.

This document is proposed by China National Light Industry Federation.

This document is under the jurisdiction of the National Liquor Standardization Technical Committee (SAC/TC 358).

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The previous versions of this document and its superseded documents are as follows:

- First published in 1989 as GB/T 10345.1-1989～GB/T 10345.8-199;
- The first revision was GB/T 10345-2007 in 2007;
- This is the second revision.,

1. Scope

This document specifies the general rules, basic requirements and detailed test procedures for liquor analysis.

This document applies to the analysis of various liquors.

2. Normative References

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, for dated references, only the version corresponding to the date applies to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 601 Chemical reagents - Preparation of standard titration solutions

Preparation of preparations and products used in GB/T 603 chemical reagents test methods

GB/T 6682-2008 Specifications and test methods for water used in analytical laboratories

3. Terms and Definitions

There are no terms and definitions that need to be defined in this document.

4. General

4.1 The terms and measurement units used in this document shall comply with the relevant national standards.

4.2 The instruments in this document are necessary for analysis, and general laboratory instruments are no longer included.

4.3 The water used in this document, the water used in the chromatographic analysis test is the water of grade 2 or above specified in GB/T 6682-2008. If no other requirements are specified, it shall comply with grade 3 or above in GB/T 6682-2008 (including tertiary) water specifications. All reagents used are of analytical grade (A.R.) unless other specifications are specified.

4.4 Solutions in this document, unless otherwise stated, refer to aqueous solutions.

4.5 The chromatographic reference conditions in the chromatographic analysis test can be based on the conditions of the instrument and chromatographic column, and the optimal chromatographic conditions can be selected through the test, so that the chromatographic peaks of the internal standard (ethanol) components and the chromatographic peaks of the components to be tested can be completely separated. The content of the component to be tested is appropriately adjusted.

4.6 After the reference substance stock solution used in this document (in which the internal standard (ethanol) reference substance stock solution is referred to as “internal standard solution” (ethanol)in this document) is prepared, transfer it to a reagent bottle and place it in a low temperature refrigerator at 0°C to 4°C and seal it. save.

4.7 When there are two or more analysis methods for the same test item, the first method shall be the arbitration method.

5. Basic requirements

5.1 To measure the samples, parallel tests should be done. The analysis results are reported as the arithmetic mean of the measured data, and do not need to be converted into alcohol content.

5.2 The significant figures in the analytical method indicate the required precision when drawing or weighing.

5.3 Constant weight means that the sample is dried and the difference between the two weighing values is below 2 mg.

6. Sensory evaluation

6.1 Principle

The tasters analyze and evaluate the color, appearance, aroma, taste, taste and style characteristics of liquor samples through the eyes, nose, mouth and other sensory organs.

6.2 Wine tasting environment

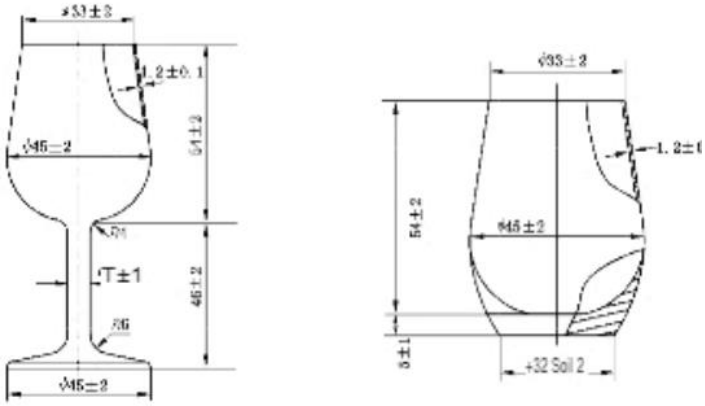
The tasting room should be well lit, soft and suitable; the temperature should be 16℃～26℃ and the relative humidity should be 30%～70%; the indoor air should be fresh, free of aroma and miscellaneous smell.

6.3 Wine evaluation requirements

6.3.1 Tasters should have sensitive sense organs, have undergone special training and assessment, meet the requirements of sensory evaluation, be familiar with the sensory evaluation terms of liquor, and master the characteristics of relevant liquor

6.3.2 Comments should be fair, scientific and accurate.

6.3.3 The dimensions of the standard wine tasting glass are shown in Figure 1. There are cup legs [see Figure 1a)] and no cup legs (see Figure 1b)], both of which are made of colorless and transparent glass, with a full capacity of 50mL to 55mL, and a maximum capacity of 15ml to 20ml at the liquid level. Conditional, the capacity scale can be added on the cup wall in millimeters



a) With cup feet b) Without cup feet

Figure 1 Schematic diagram of liquor tasting glass

6.4 Tasting

6.4.1 Preparation of samples

Put the liquor samples in the environment of 20°C to 25°C (or keep warm in a water bath at 20°C to 25°C) to equilibrate the temperature, take a password mark and conduct sensory evaluation. The amount is 15mL to 20mL.

6.4.2 Color and appearance

Pick up the wine glass, use the white wine evaluation table or white paper as the background, and observe the wine sample for color and color depth by looking straight, looking down and looking up. Then shake it gently, observe the clarity of the wine, the presence or absence of suspended matter and sediment, and record its

color and appearance.

6.4.3 Aroma

General sniffing: first lift the wine glass, place the wine glass at a slight angle of 30° at 10mm to 20mm below the nose, and lower the head slightly. Use a uniform and soothing inhalation method to smell its still aroma, and only inhale the wine when sniffing. Breath, don't breathe out. Then gently shake the wine glass to increase the volatilization and aggregation of the aroma, and then smell and record the aroma.

Special circumstances: Empty the wine and leave it for a while to smell the empty glass and leave the fragrance.

6.4.4 Taste, taste

Pour the sample into a clean and dry wine glass, drink a small amount of 0.5mL to 2.0mL of the sample in the mouth, taste it carefully with the taste organs, and note the taste and taste characteristics.

6.4.5 Style

Based on the characteristics of aroma, taste, taste, etc., combined with the characteristics of each liquor style, a summary evaluation is made to determine whether it has a typical style or a unique style (personality).

7. Total esters

7.1 Indicator method

7.1.1 Principle

Neutralize the free acid in the sample with alkali, then add a certain amount of alkali accurately, heat under reflux to saponify the esters, use standard sulfuric acid titration solution for neutralization titration, and calculate the total ester content by the amount of acid consumed.

7.1.2 Instruments

7.1.2.1 All-glass still: 500mL.
7.1.2.2 All-glass reflux device: reflux bottle 1000mL, 250mL, condenser tube not shorter than 45cm.
7.1.2.3 Basic burette: 25mL or 50mL.
7.1.2.4 Acid burette: 25mL or 50mL.

7.1.3 Reagents and solutions

7.1.3.1 Ethanol solution (95%, volume fraction): Measure 950 mL of ethanol, add 50 mL of water, and mix well.
7.1.3.2 Sodium hydroxide standard titration solution [c(NaOH)=0.1 mol/L]: Prepare and calibrate according to GB/T 601.
7.1.3.3 Sodium hydroxide solution [c(NaOH)=3.5 mol/L]: Weigh 110 g of sodium hydroxide, dissolve it in 100 mL of carbon dioxide-free water, shake it up, pour it into a polyethylene container, and place it in an airtight place until the solution is clear. Take 18.9 mL of the supernatant, dilute to 100 mL with carbon dioxide-free water, and shake well.
7.1.3.4 Standard titration solution of sulfuric acid [c(1/2H2SO2)=0.1 mol/L]: Prepare and calibrate according to GB/T 601.
7.1.3.5 Ethanol (ester-free) solution (40%, volume fraction): Measure 600 mL of 95% ethanol solution (7.1.3.1) into a 1000 mL reflux bottle, add 5 mL of sodium hydroxide solution (7.1.3.3), heat under reflux for saponification 1h. Then it was transferred to an all-glass still (7.1.2.1) for re-distillation, and then made into an ethanol (ester-free) solution (40%, volume fraction).

7.1.3.6 Phenolphthalein indicator solution (10g/L): prepared according to GB/T 603.

7.1.4 Test procedure

7.1.4.1 Draw 50.0mL of sample into a 250mL reflux bottle, add 2 drops of phenolphthalein indicator solution (7.1.3.6), and titrate with sodium hydroxide standard titration solution (7.1.3.2) to a reddish color that does not fade for 30s (do not overdo it), Record the number of milliliters of sodium hydroxide standard titration solution consumed.
7.1.4.2 Accurately add 25.00 mL of sodium hydroxide standard titration solution (7.1.3.2) (if the total ester content of the sample is high, add 50.00 mL), shake well, put in a few zeolite or glass beads, and install a condenser (The cooling water temperature should be lower than 15 ℃), refluxed on a boiling water bath for 30 min, removed and cooled.
7.1.4.3 Carry out titration with sulfuric acid standard titration solution (7.1.3.4), make the red color just completely disappear as the end point, and record the volume V1 of the consumed sulfuric acid standard titration solution. At the same time, draw 50 mL of ethanol (ester-free) solution (40%, volume fraction) (7.1.3.5), and do the same operation as above to do a blank test, and record the volume V of the standard titration solution of sulfuric acid consumed. .

7.1.5 Result calculation

The total ester content in the sample is calculated according to formula (1)

$$X_1 = \frac{c_1 \times (V_B - V_1) \times 88}{50}. \quad (1)$$

where:

X_1 – Total ester content in the sample, expressed in mass concentration (calculated in ethyl acetate), in grams per liter (g/L);

c_1 – The actual molar concentration of C1 sulfuric acid standard titration solution, the unit is moles per liter (mol/L);

V_B. – The volume of the standard titration solution of sulfuric acid consumed by the blank test sample, in milliliters (ml);

V_1 – the volume of the standard titration solution of sulfuric acid consumed by the sample, in milliliters (mL);

88 – The molar mass of ethyl acetate, in grams per mole (g/mol) [M(CH3COOC2Hs)=88];

50.0 – The volume of the aspirated sample in milliliters (mL).

Calculation results are expressed to two decimal places.

7.1.6 Precision

The absolute difference between the results of two independent determinations obtained under repeated determination conditions does not exceed 2% of the arithmetic mean.

7.2 Potentiometric titration

7.2.1 Principle

Neutralize the free acid in the sample with alkali, and then add a certain amount of alkali for reflux saponification. The neutralization titration was carried out with the standard titration solution of sulfuric acid, with pH=8.70 as the indicated end point, and the content of total esters was calculated by the consumption of the standard titration solution of sulfuric acid.

7.2.2 Instruments

7.2.2.1 All-glass still: 500mL.

7.2.2.2 All-glass reflux device: reflux bottle 1000mL, 250mL, condenser tube not shorter than 45cm.
7.2.2.3 Basic burette: 25mL or 50mL.
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7.2.2.4 Acid burette: 25mL or 50mL.
7.2.2.5 Potentiometric titrator (or acidity meter): Accuracy 0.01pH, with magnetic stirring device.
7.2.2.6 pH glass acid-base electrode.

7.2.3 Reagents and solutions

Same as 7.1.3.
7.2.4 Test procedure

7.2.4.1 Install and debug the instrument according to the instruction manual, and calibrate and position according to the solution temperature.
7.2.4.2 Draw 50.0 mL of sample into a 250 mL reflux bottle, add 2 drops of phenolphthalein indicator solution (7.1.3.6), and titrate with sodium hydroxide standard titration solution (7.1.3.2) until it is slightly red and does not fade for 30s (do not overdose), Record the number of milliliters of sodium hydroxide standard titration solution consumed.
7.2.4.3 Accurately add 25.00 mL of sodium hydroxide standard titration solution (7.1.3.2) (if the total ester content of the sample is high, add 50.00 mL), shake well, put in a few zeolite or glass beads, and install a condenser (The cooling water temperature should be lower than 15 ℃), refluxed on a boiling water bath for 30 minutes, removed and cooled. Transfer the sample solution into a 100mL small beaker, rinse the reflux bottle with 10mL water, and merge the washing solution into the small beaker.
7.2.4.4 Insert the electrode, put in a magnetic stirring bar, place it on the magnetic stirrer, and start stirring. In the initial stage, the standard titration solution of sulfuric acid (7.1.3.4) can be quickly added dropwise. When the pH of the sample solution is equal to 9.00, slow down. Titration speed, add half a drop of solution dropwise each time until pH=8.70 is the end point, record the volume V1 of the standard titration solution consumed by sulfuric acid.
7.2.4.5 Simultaneously draw 50.00 mL of ethanol (ester-free) solution (40%, volume fraction) (7.1.3.5), and perform the same operation as above to do a blank test, and record the volume V of the standard titration solution of sulfuric acid consumed.

7.2.5 Result calculation

Same as 7.1.5.

7.2.6 Precision

Same as 7.1.6.

8. Total esters

8.1 Indicator method

8.1.1 Principle

Neutralize the free acid in the sample with alkali, then add a certain amount of alkali, heat under reflux to saponify the ester, and neutralize the remaining alkali with acid. The total amount of ester is obtained by calculating the total consumption of base.

8.1.2 Instruments

Same as 7.1.2.

8.1.3 Reagents and solutions

Same as 7.1.3.

8.1.4 Test procedure

8.1.4.1 Neutralize the free acid in the sample with alkali, the test procedure is the same as 7.1.4.1, and record the volume V2 of the standard titration solution of sodium hydroxide consumed.

8.1.4.2 Heating to reflux and neutralizing the remaining alkali, the test steps are the same as 7.1.4.2~7.1.4.3, and record the volume V of the standard titration solution of sulfuric acid consumed by the blank test sample. . The sample consumes the volume V1 of the standard titration solution of sulfuric acid.

8.1.5 Result calculation

The total amount of acid ester in the sample is calculated according to formula (2).

$$X_2 = \frac{[c_2 \times V_2 + c_1 \times (V_0 - V_1) \times 1000}{50} \quad (2)$$

where:

X_2 – the total amount of esters in the sample, in millimoles per liter (mmol/L);

c_2 – the actual molar concentration of the standard titration solution of sodium hydroxide, in moles per liter (mol/L);

V_2 – the volume of sodium hydroxide standard titration solution consumed by the total acid in the sample, in milliliters (mL);

c_1 – the actual molar concentration of the standard titration solution of sulfuric acid, in moles per liter (mol/L);

V_0. – The volume of the standard titration solution of sulfuric acid consumed by the blank test sample, in milliliters (mL);

V_1 – The volume of the standard titration solution of sulfuric acid consumed by the sample, in milliliters (mL);

50.0 – The volume of the aspirated sample in milliliters (mL).

Calculation results are expressed to one decimal place.

8.1.6 Precision

Same as 7.1.6.

8.2 Potentiometric titration

8.2.1 Principle

Same as 8.1.1.

8.2.2 Instruments

Same as 7.2.2.

8.2.3 Reagents and solutions

Same as 7.2.3.

8.2.4 Test procedure

8.2.4.1 Install and debug the instrument according to the instruction manual, and calibrate the pH electrode

according to the solution temperature.
8.2.4.2 Aspirate 50.0mL of the sample (if a composite electrode is used, the sampling volume can be increased as appropriate) into a 100mL beaker, insert a pH glass acid-base electrode, put a magnetic rotor, place it on a magnetic stirring device, and start stirring. Quickly add sodium hydroxide standard titration solution (7.1.3.2), when pH=8.00, slow down the titration speed, add half a drop of solution each time until pH=9.00 is the end point, record the consumption of sodium hydroxide standard titration solution volume V2, then transfer the sample solution to a distillation flask, rinse the beaker several times with a small amount of water, and transfer it to the distillation flask.
8.2.4.3 Heating to reflux and neutralizing the remaining alkali, the test steps are the same as 7.2.4.3~7.2.4.5, and record the volume V of the standard titration solution of sulfuric acid consumed by the blank test sample. . The sample consumes the volume V1 of the standard titration solution of mineral acid.

8.2.5 Result calculation

Same as 8.1.5.

8.2.6 Precision

Same as 7.1.6.

9. Solids

9.1 Principle

After the liquor is evaporated and dried, the non-volatile substances remain in the dish, and after constant weight, it is determined by weighing method.

9.2 Instruments

9.2.1 Electric drying oven: temperature control accuracy ± 2 ℃.
9.2.2 Analytical balance: Sensitivity 0.1mg.
9.2.3 Porcelain evaporating dish or glass evaporating dish: 100mL.
9.2.4 Desiccant: use discolored silica gel as desiccant.

9.3 Test procedure

Aspirate 50.0mL of the sample, pour it into a 100mL porcelain evaporating dish or glass evaporating dish that has been dried to constant weight, place it on a boiling water bath, evaporate to dryness, and then put the evaporating dish into a (103±2) ℃ electric heating drying box, Bake for 2h, take out, put in a desiccator for 30min, and weigh. Then put it into the (103±2) ℃ electric heating drying box, bake for 1 hour, take it out, put it in the desiccator for 30 minutes, and weigh it. Repeat the above operation until constant weight.

9.4 Result calculation

The solid content in the sample is calculated according to formula (3).

$$X_3 = \frac{m\ -m_1}{50} \times 1000 \quad (3)$$

where:
X_3 – The solid content in the sample, expressed in mass concentration, in grams per liter (g/L);
m – the mass of the solids and the evaporating dish, in grams (g);
m_1 – the mass of the evaporating dish, in grams (g);
50.0 – Take the volume of the sample in milliliters (mL).
Calculation results are expressed to two decimal places.

9.5 Precision

The absolute difference between the results of two independent determinations obtained under repeated determination conditions does not exceed 5% of the arithmetic mean.

10. Ethyl acetate, ethyl butyrate, ethyl hexanoate, ethyl lactate, n-propanol, β-phenethyl alcohol

10.1 Principle

After the sample is vaporized, it is separated by a chromatographic column. Since the components to be measured have different distribution coefficients in the gas-liquid two-phase, the separated. The measured components flow out of the chromatographic column in sequence and enter the hydrogen flame ionization detector for detection. According to the retention value of each component peak on the chromatogram compared with that of the standard substance, the quantification is carried out, using the peak area (or peak height), the internal standard (ethanol) method Quantitative.

10.2 Reagents and materials

10.2.1 Ethanol: chromatographically pure.
10.2.2 Standard substances such as ethyl acetate, ethyl butyrate, ethyl hexanoate, ethyl lactate, n-propanol, β-phenethyl alcohol, etc.: purity ≥ 99%, or standard substances certified by the state and granted a standard substance certificate .
10.2.3 Standard substances of tert-amyl alcohol, n-amyl acetate and n-butyl acetate: the purity is ≥99%, or the standard substance certified by the state and granted the certificate of standard substance, used as the internal standard.
10.2.4 Ethanol solution (50%, volume fraction): Measure 250 mL of ethanol (10.2.1), add 250 mL of water, and mix well.
10.2.5 n-propanol standard substance stock solution (10000mg/L): accurately weigh 1.0g (accurate to 1mg) n-propanol standard substance
(10.2.2), add an appropriate amount of ethanol solution (50%, volume fraction) (10.2.4) to dissolve, transfer to a 100mL volumetric flask, make up to volume, and mix well.
10.2.6 β-phenethyl alcohol standard stock solution (500mg/L): accurately weigh 0.05g (accurate to 1 mg) β-phenethyl alcohol standard material (10.2.2), add an appropriate amount of ethanol solution (50%, volume fraction)) (10.2.4) to dissolve, transfer to a 100mL volumetric flask, make up to volume, and mix well.
10.2.7 Mixed stock solution of ester standard substances (25000mg/L for ethyl acetate, ethyl hexanoate and ethyl lactate, and 2500mg/L for ethyl butyrate): respectively accurately weigh 2.50g (accurate to 1mg) Ethyl acetate standard substance (10.2.2), ethyl hexanoate standard substance (10.2.2), ethyl lactate standard substance (10.2.2), 0.25g (accurate to 1mg) ethyl butyrate standard substance (10.2. 2), add an appropriate amount of ethanol (10.2.1) to dissolve, transfer to a 100mL volumetric flask, make up to volume, and mix well.
10.2.8 Mixed internal standard (ethanol) solution of tert-amyl alcohol and n-amyl acetate (20000 mg/L): used as internal standard (ethanol) when using capillary chromatographic column. Accurately weigh 2.0g (accurate to 1mg) of tert-amyl alcohol standard substance (10.2.3) and n-amyl acetate standard substance (10.2.3), add an appropriate amount of ethanol solution (50%, volume fraction) (10.2.4) to dissolve, transfer to a 100mL volumetric flask, make up to volume, and mix thoroughly.
10.2.9 n-Butyl acetate internal standard (ethanol) solution (20000mg/L): used as internal standard (ethanol) when using packed chromatographic column. Accurately weigh 2.0g (accurate to 1mg) of n-butyl acetate standard substance (10.2.3), add an appropriate amount of ethanol solution (50%, volume fraction) (10.2.4) to dissolve, transfer to a 100mL volumetric flask, and set the volume , and mix thoroughly.
10.2.10 Mixed standard working solution 1 of esters and alcohols series (applicable to 10.4.1.1): draw 0.1mL, 0.2mL, 0.4mL, 0.6mL, 1.0mL of n-propanol standard material stock solution (10.2.5) respectively , β-phenethyl alcohol standard material stock solution (10.2.6) and ester standard material mixed stock solution (10.2.7) in five 10mL volumetric flasks, and then add 0.1mL tert-amyl alcohol, n-amyl acetate internal standard (ethanol) respectively Solution (10.2.8), use ethanol solution (50%, volume fraction)

(10.2.4) to volume, and mix well. Formulated into ethyl acetate, ethyl caproate, ethyl lactate: 250 mg/L, 500 mg/L, 1 000 mg/L, 1 500 mg/L, 2 500 mg/L, ethyl butyrate: 25 mg/L , 50 mg/L, 100 mg/L, 150 mg/L, 250 mg/L, n-propanol is 100 mg/L, 200 mg/L, 400 mg/L, 600 mg/L, 1 000 mg/L, β-phenethyl alcohol It is a series of mixed standard working solutions of 5mg/L, 10mg/L, 20mg/L, 30 mg/L and 50mg/L, which are prepared and used now.

10.2.11 Mixed standard working solution 2 of esters and alcohols series (applicable to 10.4.1.2): draw 0.1mL, 0.2mL, 0.4mL, 0.6mL, 1.0mL of n-propanol standard material stock solution (10.2.5) respectively , Ester standard substance stock solution (10.2.7) in 5 10mL volumetric flasks, then add 0.1mL n-butyl acetate internal standard (ethanol) solution (10.2.9) respectively, use ethanol solution (50%, volume fraction) (10.2.4) Set the volume and mix well. Formulated into ethyl acetate, ethyl caproate, ethyl lactate: 250mg/L, 500 mg/L, 1000 mg/L, 1500 mg/L, 2 500 mg/L, ethyl butyrate: 25 mg/L, 50 mg/L , 100 mg/L, 150 mg/L, 250 mg/L, n-propanol is 100 mg/L, 200 mg/L, 400 mg/L, 600 mg/L, 1000 mg/L series of mixed standard working solutions. With current use.

10.3 Instruments and equipment

10.3.1 Gas chromatograph equipped with hydrogen flame ionization detector (FID).

10.3.2 Analytical balance: the sensitivity is 0.1 mg.

10.3.3 Pipette: 0.1mL～1.0mL.

10.4 Test procedure

10.4.1 Chromatographic reference conditions

10.4.1.1 Capillary column

10.4.1.1.1 Polyethylene glycol capillary column (60mx0.25mmx0.25μm) or (50mx0.25mmx0.20μm) or other chromatographic column with equivalent analytical effect.

10.4.1.1.2 Heating program: the initial temperature is 35°C, hold for 1min, rise to 70°C at 3.0°C/min, rise to 180°C at 3.5°C/min, and then rise to 210°C at 15°C/min, hold for 6min.

10.4.1.1.3 Detector temperature: 250°C.

10.4.1.1.4 Inlet temperature: 250°C.

10.4.1.1.5 Constant flow mode: 1.0 mL/min.

10.4.1.1.6 Injection volume: 1.0 μL.

10.4.1.1.7 Split ratio: 40:1.

10.4.1.2 Packing the column

10.4.1.2.1 Packed column: the column length is not less than 2m.

10.4.1.2.2 Carrier: Chromosorb W (AW) or white carrier 102 (pickling, silanization), 80-100 mesh (0.18mm-0.125mm).

10.4.1.2.3 Fixative: 20% dinonyl phthalate (DNP) plus 7% Tween 80, or 10% polyethylene glycol (PEG) 1500 or PEG 20 mol/L.

10.4.1.2.4 Carrier gas (high-purity nitrogen): the flow rate is 150mL/min.

10.4.1.2.5 Hydrogen: the flow rate is 40mL/min.

10.4.1.2.6 Air: the flow rate is 400mL/min.

10.4.1.2.7 Detector temperature: 150°C.

10.4.1.2.8 Inlet temperature: 150°C.

10.4.1.2.9 Column temperature: 90°C, isothermal.

10.4.2 Plotting the standard curve

10.4.2.1 Capillary column

Pipette an appropriate amount of mixed standard working solution 1 (10.2.10) of the series of esters and alcohols, inject and measure according to the chromatographic reference conditions (10.4.1.1), and use the retention of the chromatographic peaks of each ester and alcohol component as a single standard Time is used as the basis for qualitative determination. The ratio of the concentration of each ester and alcohol to the corresponding internal standard concentration is the abscissa, and the ratio of the peak area of each ester and alcohol component to the corresponding internal standard (ethanol) peak area is the ordinate, where n-propyl For alcohol and β-phenethyl alcohol, use tert-amyl alcohol as internal standard (ethanol); for ethyl acetate, ethyl butyrate, ethyl hexanoate, and ethyl lactate, use n-amyl acetate as internal standard (ethanol); draw standard working curve.

10.4.2.2 Packing the column

Pipette an appropriate amount of ester and alcohol series mixed standard working solution 2 (10.2.11), inject and measure according to the chromatographic reference conditions (10.4.1.2), and use the retention of the chromatographic peaks of each ester and alcohol component as a single standard Time is used as the basis for qualitative determination. The ratio of the concentration of each ester and alcohol to the internal standard concentration of n-butyl acetate is the abscissa, and the ratio of the peak area of each ester and alcohol component to the internal standard (ethanol) peak area of n-butyl acetate is Ordinate, draw standard working curve.

10.4.3 Sample determination

Pipette an appropriate amount of sample into a 10mL volumetric flask, add 0.1mL of the internal standard (ethanol) solution (10.2.8 or 10.2.9), use the same sample to dilute to volume, mix thoroughly, and measure the sample according to the chromatographic reference conditions (10.4.1). The ratio I1 of the mass concentration of each component to be tested in the sample to the mass concentration of the corresponding internal standard (ethanol) is obtained from the standard working curve, and then the content of each component to be tested in the sample is calculated according to the concentration pi of the internal standard (ethanol) corresponding to the component to be tested.

10.5 Result calculation

10.5.1 The content of ethyl acetate, ethyl butyrate, ethyl hexanoate, ethyl lactate and n-propanol in the sample is calculated according to formula (4).

(4)

$$X_i = \frac{l_i \times p_i}{1000} \tag{4}$$

where:

X_i – the content of ethyl acetate, ethyl butyrate, ethyl hexanoate, ethyl lactate and n-propanol in the sample, expressed in mass concentration, in grams per liter (g/L);

l_i – obtain the ratio of ethyl acetate, ethyl butyrate, ethyl hexanoate, ethyl lactate, n-propanol concentration and corresponding internal standard (ethanol) concentration in the liquid to be tested from standard curve;

p_i – The mass concentration of the internal standard (ethanol) corresponding to ethyl acetate, ethyl butyrate, ethyl hexanoate, ethyl lactate and n-propanol, in milligrams per liter (mg/L);

1000 – Unit conversion factor.

Calculation results are expressed to two decimal places.

10.5.2 The content of β-phenethyl alcohol in the sample is calculated according to formula (5).

$$X_j = l_j \times p_j \tag{5}$$

X_j – phenethyl alcohol - the content of β-phenethyl alcohol in the sample, expressed in mass concentration, in milligrams per liter (mg/L);

l_j – 18 m ethanol-obtain the ratio of the mass concentration of test solution β-phenethyl alcohol and the internal standard (ethanol) mass concentration of tert-amyl alcohol from the standard curve;
p_j – Mass concentration of PB phenethyl alcohol-tert-amyl alcohol internal standard (ethanol), in milligrams per liter (mg/L).
The calculation result is expressed as an integer.

10.6 Precision

The absolute difference between the results of two independent determinations obtained under repeated determination conditions does not exceed 5% of the arithmetic mean.

11. Acetic acid

11.1 Principle

Same as 10.1.

11.2 Reagents and Materials

11.2.1 Ethanol: chromatographically pure.
11.2.2 Acetic acid reference material: purity ≥99%, or a reference material certified by the state and granted a reference material certificate.
11.2.3 2-Ethylbutyric acid standard substance: purity ≥99%, or a standard substance certified by the state and granted a standard substance certificate, used as an internal standard (ethanol) .
11.2.4 Ethanol solution (50%, volume fraction): Same as 10.2.4.
11.2.5 Acetic acid standard material stock solution (20000 mg/L): accurately weigh 2.0 g (accurate to 1 mg) of acetic acid standard material
(11.2.2), add an appropriate amount of ethanol solution (50%, volume fraction) (11.2.4) to dissolve, transfer to a 100mL volumetric flask, make up to volume, and mix well.
11.2.6 2-ethylbutyric acid internal standard (ethanol) solution (20 000 mg/L): Weigh 2.0g (accurate to 1mg) 2-ethylbutyric acid standard substance
(11.2.3), add an appropriate amount of ethanol solution (50%, volume fraction) (11.2.4) to dissolve, transfer to a 100mL volumetric flask, make up to volume, and mix well.
11.2.7 Acetic acid series standard working solution: accurately pipette 0.2mL, 0.4mL, 0.6mL, 0.8mL, 1.0mL acetic acid standard stock solution (11.2.5) into a 10mL volumetric flask, and then add 0.1mL 2-ethyl acetate respectively Butyric acid internal standard (ethanol) solution (11.2.6), with ethanol solution (50% by volume) (11.2.4)
Dilute to volume, shake well, and prepare standard working solutions of acetic acid series of 400 mg/L, 800 mg/L, 1 200 mg/L, 1 600 mg/L and 2 000 mg/L, which are prepared and used now.

11.3 Instruments and Equipment

Same as 10.3.

11.4 Test Procedure

11.4.1 Chromatographic reference conditions

Same as 10.4.1.1.

11.4.2 Plotting the standard curve

Pipette an appropriate amount of the standard working solution of acetic acid series (11.2.7) and measure it

according to the chromatographic reference conditions (11.4.1), take the ratio of the concentration of the standard working solution of acetic acid series to the concentration of 2-ethylbutyric acid internal standard (ethanol) solution as the abscissa, The ratio of the peak area of the acetic acid series standard working solution to the peak area of the 2-ethylbutyric acid internal standard solution (ethanol) is drawn as the ordinate to draw a standard curve.

11.4.3 Sample determination

Pipette an appropriate amount of sample into a 10mL volumetric flask, add 0.1mL 2-ethylbutyric acid internal standard solution (11.2.6), use the same sample to make up the volume, and mix well. Measured according to the chromatographic reference conditions (11.4.1), according to the retention time of the acetic acid standard substance, and qualitatively with the retention time of acetic acid in the sample to be tested. According to the ratio of the peak areas of acetic acid and 2-ethylbutyric acid internal standard solution (ethanol) in the test solution, the ratio of the concentration of acetic acid and 2-ethylbutyric acid internal standard solution in the test solution was obtained from the standard working curve, and then according to the 2-The concentration of ethyl butyric acid internal standard solution was used to calculate the content of acetic acid in the sample.

11.5 Result calculation

Same as 10.5.1.

11.6 Precision

The absolute difference between the results of two independent determinations obtained under repeated determination conditions does not exceed 10% of the arithmetic mean.

12. Caproic acid

12.1 Gas chromatography

12.1.1 Principle

Same as 10.1.

12.1.2 Reagents
12.1.2.1 Ethanol: chromatographically pure.
12.1.2.2 Caproic acid reference material: purity ≥99.5%, or a reference material certified by the state and granted a reference material certificate.
12.1.2.3 2-Ethylbutyric acid reference material: the purity is ≥99%, or the reference material certified by the state and granted with a reference material certificate, is used as an internal standard.
12.1.2.4 Ethanol solution (50%, volume fraction): same as 10.2.4.
12.1.2.5 Caproic acid standard substance stock solution (10000mg/L): Weigh 1.0g (accurate to 1mg) caproic acid standard substance (12.1.2.2) and add an appropriate amount of ethanol solution (50%, volume fraction) (11.2.4) to dissolve , transfer to a 100mL volumetric flask, make up to volume, and mix well.
12.1.2.6 2-ethylbutyric acid internal standard solution (20 000mg/L) (ethanol) : same as 11.2.6.
12.1.2.7 Caproic acid series standard working solution: accurately pipette 0.2mL, 0.4mL, 0.6mL, 0.8mL, 1.0mL caproic acid standard stock solution (12.1.2.5) into a 10mL volumetric flask, and then add 0.1mL of 2 - Ethyl butyric acid internal standard (ethanol) solution (12.1.2.6), use ethanol solution (50%, volume fraction) (12.1.2.4) to make up the volume, and mix well. Prepared into 200 mg/L, 400 mg/L, 600 mg/L, 800 mg/L, 1000 mg/L caproic acid series standard working solutions, which are prepared and used now.

12.1.3 Instruments and equipment

Same as 10.3.

12.1.4 Test procedure

12.1.4.1 Chromatographic reference conditions

Same as 10.4.1.1.

12.1.4.2 Plotting the standard curve

Except that the standard substance is changed to hexanoic acid series standard working solution (12.1.2.7), other operations are the same as 11.4.2.

12.1.4.3 Sample determination

Except that the standard substance is changed to hexanoic acid series standard working solution (12.1.2.7), other operations are the same as 11.4.3.

12.1.5 Result calculation

Same as 10.5.1.

12.1.6 Precision

Same as 11.6.

12.2 Ion chromatography

12.2.1 Principle

After the sample was diluted with water, it was separated by anion exchange chromatographic column and measured by conductivity detector. It was qualitative by retention time and quantified by external standard method.

12.2.2 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade, and the water is first-grade water specified in GB/T 6682-2008.

12.2.2.1 Ethanol: chromatographically pure.
12.2.2.2 Sodium carbonate.
12.2.2.3 Sodium bicarbonate.
12.2.2.4 Sodium hydroxide.
12.2.2.5 Caproic acid reference material: same as 12.1.2.2.
12.2.2.6 Sodium carbonate solution (1mmol/L): Accurately weigh 0.106g of sodium carbonate solution, dissolve it with an appropriate amount of water, dilute to 1000mL, and mix well.
12.2.2.7 Mixed solution of sodium carbonate and sodium bicarbonate (sodium carbonate 13mmol/L, sodium bicarbonate 2mmol/L): accurately weigh 1.378g sodium carbonate solution (12.2.2.6) and 0.168g sodium bicarbonate (12.2.2.3) , dissolve with appropriate amount of water, make up to 1000mL, and mix well
12.2.2.8 Sodium hydroxide solution (100 mmol/L): Weigh 4.0 g of sodium hydroxide (12.2.2.1), dissolve it with an appropriate amount of water, dilute to 1000 mL, and mix well. Can also be prepared using an automatic eluent generator OH-type
12.2.2.9 Ethanol solution (50%, volume fraction): same as 10.2
12.2.2.10 Caproic acid standard stock solution (1.000mg/L) Weigh 0.1g (accurate to 1mg) caproic acid

standard substance (12.2.2.5) and add appropriate amount of ethanol solution (50%, volume fraction) (12.2.2.9) to dissolve , transfer to a 100mL volumetric flask, make up to volume, and mix well.
12.2.2.11 Caproic acid series standard working solution; Pipette appropriate amount of caproic acid standard stock solution (12.2.2.10) and prepare with water to prepare 5.0 mg/L, 10.0 mg/L, 15.0 mg/L, 20.0 mg/L, 30.0 mg/L mmg/l. A series of standard working solutions are prepared and used now.

12.2.3 Instruments and equipment

12.2.3.1 Ion chromatograph: with conductivity detector and gradient elution system.
12.2.3.2 Analytical balance: 0.1 mg inductance.
12.2.3.3 Organic microporous membrane: 0.45μm.

12.2.4 Test procedure

12.2.4.1 Sample Preparation

Accurately pipette 1.0mL of the sample into a 50mL volumetric flask, dilute to volume with water, and mix evenly; the diluted sample is filtered with an organic microporous membrane, and 1mL of the filtered sample is collected in a sample bottle for testing.

12.2.4.2 Chromatographic reference conditions

12.2.4.2.1 Carbonate/bicarbonate elution system

The chromatographic reference conditions of carbonate/bicarbonate elution system are as follows:

- Anion exchange column chromatography: polystyrene and divinylbenzene copolymer filler, quaternary ammonium salt active group 4.0mmx250mm (with the same type of guard column 4.0mmx5mm), or ion chromatography column with equivalent performance;
- Eluent: A is sodium carbonate solution (12.2.2.6), B is a mixed solution of sodium carbonate and sodium bicarbonate (12.2.2.7);
- Suppressor: ultra-fine filled inlay structure;
- Flow rate: 0.7mL/min;
- Column oven temperature: room temperature;
- Injection volume: 20 μL;
- Detector: Conductivity detector;
- Detection cell temperature: 35℃;
- The gradient elution procedure is shown in Table 1.

Table 1 Gradient elution procedure

serial number	time／min	A/%	B/%
1	0	100	0
2	18	100	0
3	18	0	100
4	23	0	100
5	23	100	0
6	45	100	0

12.2.4.2.2 Hydroxide Elution System

- The chromatographic reference conditions of the hydroxide elution system are as follows:

- Anion chromatography column: Divinylbenzene-ethylvinylbenzene copolymer packing, alkanol quaternary ammonium salt exchange functional group, 4.0mmx250mm (with the same type of guard column 4.0mmx5mm), or an ion chromatography column with equivalent performance;
- Eluent: produced by the on-line generator of hydroxide eluent;
- Suppressor: self-circulation mode or equivalent performance suppressor, suppressing current 93mA;
- Flow rate: 1.5 mL/min;
- Column oven temperature: 35℃;
- Injection volume: 20 μL;
- Detector: Conductivity detector;
- Detection cell temperature: 35℃;
- The gradient elution procedure is shown in Table 2.

Table 2 Gradient elution procedure

serial number	time／min	Elution concentration／（mmol／L）
1	0	5
2	15	5
3	20	60
4	25	60
5	26	5
6	33	5

12.2.4.3 Standard curve plotting

Take the hexanoic acid series standard working solution (12.2.2.11), measure it according to the chromatographic reference conditions (12.2.4.2), use the retention time of the hexanoic acid chromatographic peak as the basis for qualitative determination, and take the concentration of the hexanoic acid series standard working solution as the abscissa , and draw a standard curve with the peak area as the ordinate.

12.2.4.4 Sample determination

Inject the prepared sample (12.2.4.1) into the ion chromatograph, measure the caproic acid chromatographic peak area according to the chromatographic reference conditions (12.2.4.2), and obtain the caproic acid content in the test solution according to the standard working curve.

12.2.4.5 Result calculation

The content of hexanoic acid in the sample is calculated according to formula (6).

$$\boldsymbol{X_1 = \frac{p_1}{1000} \times n} \quad \textbf{(6)}$$

where:
$\boldsymbol{X_1}$ – the content of hexanoic acid in the sample, expressed in mass concentration, in grams per liter (g/L);
$\boldsymbol{p_1}$ – The mass concentration of hexanoic acid in the solution to be tested is obtained from the standard curve, in milligrams per liter (mg/L)
$\boldsymbol{n}$ – sample dilution factor.
Calculation results are expressed to two decimal places.

12.2.5 Precision

Same as 11.6.

13. Ethyl propionate

13.1 Principle

Same as 10.1.

13.2 Reagents and Materials

13.2.1 Ethanol: chromatographically pure.
13.2.2 Ethyl propionate reference material: purity ≥99%, or a reference material certified by the state and granted a reference material certificate.
13.2.3 Standard substance of n-amyl acetate: purity ≥99%, or a standard substance certified by the state and granted a certificate of standard substance, used as an internal standard (ethanol) .
GB/T 10345-2022
13.2.4 Ethanol solution (50%, volume fraction): Same as 10.2.4.
13.2.5 n-amyl acetate internal standard (ethanol) solution (10000 mg/L): Weigh 0.5g (accurate to 1 mg) n-amyl acetate standard substance
(13.2.3), add an appropriate amount of ethanol solution (50%, volume fraction) (13.2.4) to dissolve, transfer to a 50mL volumetric flask, make up to volume, and mix well.
13.2.6 Standard material stock solution of ethyl propionate (1000mg/L): accurately weigh 0.1g (accurate to 1mg) of ethyl propionate standard material (13.2.2), add an appropriate amount of ethanol solution (50%, volume fraction)) (13.2.4) to dissolve, transfer to a 100mL volumetric flask, make up to volume, and mix thoroughly.
13.2.7 Standard working solution of ethyl propionate series: pipette 0.05mL, 0.1mL, 0.2mL, 0.3mL, 0.4mL stock solution of ethyl propionate (13.2.6) into five 10mL volumetric flasks, respectively. Add 0.1 mL of n-amyl acetate internal standard (ethanol) solution (13.2.5), dilute to volume with ethanol solution (50%, volume fraction) (13.2.4), mix well, and prepare 5.0 mg/L and 10.0 mg/L in turn , 20.0 mg/L, 30.0 mg/L, 40.0 mg/L series of standard working solutions are now prepared and used.

13.3 Instruments and Equipment

Same as 10.3.

13.4 Test Procedure

13.4.1 Chromatographic reference conditions
13.4.1.1 Medium polarity stationary phase 6% cyanopropylbenzene, 94% dimethylsiloxane (30mx0.53mmx3.0μm) or other chromatographic column with the same analytical effect.
13.4.1.2 Chromatographic column temperature: the initial temperature is 35°C, hold for 8min, rise to 160°C at 10.0°C/min, and hold for 5min.
13.4.1.3 Detector temperature: 230°C.
13.4.1.4 Inlet temperature: 230°C.
13.4.1.5 Carrier gas flow rate: 2.0mL/min.
13.4.1.6 Injection volume: 1.0 μL.
13.4.1.7 Split ratio: 30:1.

13.4.2 Plotting the standard curve

Pipette an appropriate amount of the standard working solution of ethyl propionate series (13.2.7), inject and measure according to the chromatographic reference conditions (13.4.1), and use the retention time of the

ethyl propionate standard chromatographic peak as the basis for qualitative determination. The ratio of the concentration of ethyl acetate to the concentration of the internal standard (ethanol) solution of n-amyl acetate is the abscissa, the ratio of the peak area of ethyl propionate to the peak area of the internal standard solution (ethanol) of n-amyl acetate is the ordinate, and the standard working curve is drawn.

13.4.3 Sample determination

Pipette an appropriate amount of sample into a 10mL volumetric flask, add 0.1mL n-amyl acetate internal standard (ethanol) solution (13.2.5), use the same sample to make up the volume, and mix well. According to the chromatographic reference conditions (13.4.1), determine the peak area of ethyl propionate and the internal standard (ethanol) solution of n-amyl acetate in the solution to be tested. The ratio I of ethyl propionate and n-amyl acetate internal standard (ethanol) solution concentration in the solution to be tested is obtained from the standard working curve, and then according to the mass concentration p2 of n-amyl acetate internal standard (ethanol) solution, calculate the ethyl propionate in the sample content.

13.5 Result calculation

The content of ethyl propionate in the sample is calculated according to formula (7).

$$X_5 = I \times p_2 \quad (7)$$

where:
X_5 – The content of ethyl propionate in the sample, expressed in mass concentration, in milligrams per liter (mg/L);
I – obtain the ratio of the mass concentration of ethyl propionate and the internal standard (ethanol) mass concentration of n-amyl acetate in the liquid to be tested from the standard curve;
p_2 – The mass concentration of the internal standard (ethanol) P2-n-amyl acetate, in milligrams per liter (mg/L).
Calculation results are expressed to one decimal place.

13.6 Precision

Same as 10.6.

14. Diethyl ester of dibasic acid (pimelic acid, suberic acid, azelaic acid)

14.1 Principle

Same as 10.1.

14.2 Reagents and Materials

14.2.1 Ethanol: chromatographically pure.
14.2.2 Standard substances of diethyl pimelic acid, diethyl suberate, and diethyl azelaic acid: the purity is ≥99%, or the standard substances certified by the state and granted the certificate of standard substance.
14.2.3 Myristyl alcohol reference material: purity ≥99%, or a reference material certified by the state and granted a reference material certificate, used as an internal standard (ethanol) .
14.2.4 Ethanol solution (30%, volume fraction): Measure 30 mL of ethanol, add 70 mL of water, and mix well.
14.2.5 Diethyl pimelic acid, diethyl suberic acid, and diethyl azelaic acid mixed standard material stock solution (200 mg/L): Weigh 0.02 g (accurate to 1 mg) of pimelic acid diethyl ester respectively Ethyl ester, diethyl suberate, and diethyl azelaate standard substances (14.2.2), add ethanol solution (30%, volume fraction) (14.2.4) to dissolve, transfer to a 100mL volumetric flask, and make up to volume , and mix thoroughly.
14.2.6 Internal standard (ethanol) solution of tetradecanol (1000mg/L): Weigh 0.1g (accurate to 1mg) of

tetradecanol standard substance (14.2.3), add appropriate amount of ethanol (14.2.1) to dissolve, and transfer to 100mL In a constant volume bottle, set the volume and mix thoroughly.
14.2.7 Intermediate solution of mixed standard material of diethyl pimelic acid, diethyl suberate and diethyl azelaic acid (20mg/L): draw 1 mL of diethyl pimelic acid and diethyl suberate , Diethyl azelaic acid mixed standard material stock solution (14.2.5), in a 10mL volumetric flask, dilute to volume with ethanol solution (30%, volume fraction) (14.2.4), and mix well.
14.2.8 Working solution of diethyl pimelic acid, diethyl suberate and diethyl azelaic acid series mixed standard substances: draw 0.2mL, 0.4mL, 0.6mL, 1.0mL and 2.0mL of pimelic acid respectively Diethyl ester, diethyl suberate, diethyl azelaic acid mixed standard material intermediate solution (14.2.7), into five 10mL volumetric flasks, respectively add 0.1mL of tetradecanol internal standard (ethanol) solution (14.2.6), use ethanol solution (30%, volume fraction) (14.2.4) to make up the volume, mix well, and prepare 0.4 mg/L, 0.8 mg/L, 1.2 mg/L, 2.0 mg/L, 4.0 mg/L in turn. L series mixed standard working solution, ready to use.

14.3 Instruments and Equipment

Same as 10.3.

14.4 Test Procedure

14.4.1 Chromatographic reference conditions

14.4.1.1 Chromatographic reference conditions 1

Same as 10.4.1.1.

14.4.1.2 Chromatographic reference conditions 2

14.4.1.2.1 Chromatographic column: polyethylene glycol gas chromatography column (30mx0.32mmx0.25μm) or equivalent column.
14.4.1.2.2 Chromatographic column temperature: initial temperature of 50°C, hold for 3min, rise to 170°C at 10.0°C/min, then rise to 180°C at 5°C/min, hold for 5min, then rise to 210 at 20°C/min ℃ for 3 min.
14.4.1.2.3 Detector temperature: 250°C.
14.4.1.2.4 Inlet temperature: 250°C.
14.4.1.2.5 Constant Pressure Mode—Column Pressure: 82.74 kPa (12 psi).
14.4.1.2.6 Injection volume: 1.0 μL.
14.4.1.2.7 Split ratio: 20:1.

14.4.2 Plotting the standard curve

Pipette an appropriate amount of diethyl pimelic acid, diethyl suberic acid, and diethyl azelaic acid series mixed standard working solution (14.2.8), inject and measure according to the chromatographic reference conditions (14.4.1) The chromatographic peaks of diethyl diethyl subate, diethyl suberate and diethyl azelaic acid were used as the basis for the characterization of the chromatographic peaks. The ratio of the concentration of diethyl ester to the concentration of tetradecanol internal standard (ethanol) solution is the abscissa, the peak area of diethyl pimelic acid, diethyl suberate, and diethyl azelaic acid and the peak area of tetradecanol internal standard (ethanol) solution The ratio is the ordinate, and the standard working curve is drawn.

14.4.3 Sample determination

Pipette an appropriate amount of sample into a 10mL volumetric flask, add 0.1mL of tetradecanol internal standard (ethanol) solution (14.2.6), use the same sample to make up the volume, and shake well. According to the chromatographic reference conditions (14.4.1), determine the peak areas of diethyl pimelic acid, diethyl suberate, and diethyl azelaic acid and the peak area of tetradecanol internal standard (ethanol) in the liquid to be tested. According to the ratio of the internal standard peak areas of diethyl pimelic acid, diethyl suberate, diethyl

azelaate and tetradecanol in the liquid to be tested, the standard working curve was used to obtain diethyl pimelic acid in the liquid to be tested. The ratio I of the concentration of ester, diethyl suberate, diethyl azelaate and tetradecanol internal standard (ethanol) solution, and then according to the concentration of the tetradecanol internal standard standard (ethanol) solution, calculate the diethyl pimelic acid, Contents of diethyl suberate and diethyl azelaate.

14.5 Result calculation

14.5.1 The content of diethyl pimelic acid, diethyl suberate and diethyl azelaic acid in the sample is calculated according to formula (8).

$$X_6 = I_i \times p_3 \quad (8)$$

where:

X_6 – The content of diethyl pimelic acid, diethyl suberate, and diethyl azelaic acid in the sample, expressed in mass concentration, in milligrams per liter (mg/L);

I_i – obtain the ratio of the mass concentration of diethyl pimelic acid, diethyl suberate, and diethyl azelaic acid to the internal standard (ethanol) mass concentration of tetradecanol in the liquid to be tested from the standard curve;

p_3 – The mass concentration of the internal standard (ethanol) of P3-tetradecanol, in milligrams per liter (mg/L).

Calculation results are expressed to two decimal places.

14.5.2 The total amount of diethyl dibasic acid in the sample is calculated according to formula (9).

$$X_7 = \sum X_i \quad (9)$$

where:

X_7 – The total amount of diethyl esters of dibasic acids (pimelic acid, suberic acid, azelaic acid) in the sample, expressed in mass concentration, in milligrams per liter (mg/L);

X_i – The content of diethyl pimelic acid, diethyl suberic acid, and diethyl azelaic acid in the sample, the unit is milligram per liter (mg/L). Calculation results are expressed to one decimal place.

14.6 Precision

The absolute difference between the results of two independent determinations obtained under repeatability conditions does not exceed 10% of the arithmetic mean.

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